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DETERIORATION OF RUBBER AND PLASTIC INSULATION BY DEEP-OCEAN MICROORGANISMS

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U. S. NAVAL CIVIL ENGINEERING LABORATORY

Port Hueneme, California

DETERIORATION OF RUBBER AND PLASTIC INSULATION BY DEEP-OCEAN MICROORGANISMS

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Type B Final Report

by

J. S. Muraoka

ABSTRACT

This laboratory research study determined the relative deteriorating effects of deep-ocean microorganisms on five electrical insulating materials. The plastic and rubber materials were exposed for 21 months in (1) sea water containing microorganisms from deep-ocean sediments or (2) deep-ocean sediment. Control specimens were exposed in sterile sea water. Relative values for deterioration of the insulating materials were determined on the basis of insulation resistance and voltage breakdown tests. Other parameters of the deep-ocean environment, such as high hydrostatic pressures, low temperatures, and low dissolved oxygen were not a part of this study but will be considered in a future study.

Of the five materials, neoprene rubber was highly resistant to water absorption in the absence of microbes but was very susceptible to microbial deterioration. Polyethylene was highly resistant to microbes, but after 14 months it was very susceptible to water absorption. Silicone rubber, GR-S rubber, and polyvinyl chloride were fairly resistant to both microbes and water.

INTRODUCTION

The potential importance of the sea to man's existence has resulted in the rapid expansion of his interest in marine sciences and deep-ocean engineering during recent years. As marine engineering and oceanographic investigations increase, it becomes essential to know more about the effects of a sea water environment on materials and equipment. Studies have shown that rubber products,[1,2] hydrocarbons,[3] and certain synthetic organic compounds[4,5] are utilized by marine bacteria, and that various rubber electrical insulating materials[6] and pipe and cable coatings[7] are vulnerable to soil bacteria and fungi. Microbial decomposition of these products are accomplished by oxidation of hydrocarbons and are utilized as a source of carbon for the growth and multiplication of microorganisms. In a laboratory study,[2] it was definitely proved that strains of Streptomyces were able to utilize rubber hydrocarbons and that these strains produced visible holes and pits on thin rubber strips.

Because very little is known about the behavior of materials exposed in the deep-sea environment, the present study was undertaken to see what effect deep-ocean microorganisms would have upon various kinds of electrical insulating materials.

Although exposed to deep-sea sediment and microorganisms, the insulating materials were not subjected to the combined effects of high hydrostatic pressures, low dissolved oxygen concentration, low temperatures, and other parameters of a deep-ocean environment. NCEL is now fabricating six 9-inch-ID dynamic pressure vessels for testing materials in simulated deep-ocean conditions.

MATERIALS AND METHODS

Five electrical insulating materials were exposed to deep-sea microorganisms: polyethylene, polyvinyl chloride (PVC), neoprene, silicone rubber, and GR-S rubber. Table I lists the basic materials used in their formulation. These materials were 0.015 inch (15 mils) thick and covered a No. 16 tin-coated copper conductor. Four specimens of each material were randomly selected. They were 15 inches long, and 12 inches of this was exposed to the test medium. Of the four specimens, two were stressed (coiled) and two were nonstressed (straight). Stress was applied by coiling a 15-inch specimen lightly around a 1/4-inch-diameter glass rod and then removing the rod. One end of each specimen was sealed with two coats of rubber cement; the opposite end was left exposed for electrical connection. Duplicate specimens,

stressed and nonstressed, of each of the five materials were placed in 4- by 4- by 12-inch-ID sterile acrylic containers with a lid (Figure 1). An acrylic tube, one end of which was plugged with cotton, was attached to the lid as an air vent. Epoxy sealed the area between the conductors and the acrylic containers. A 1/8-inch-diameter stainless steel rod was placed longitudinally through the acrylic box, with both ends protruding. The stainless steel rod was used as an electrode.

To determine whether insulating materials would fail from microbial activity or sea water absorption, the stressed and nonstressed test specimens were exposed to the following environments (Figure 2):

1. Sea water sterilized in an autoclave
2. Sea water containing deep-sea microorganisms (deep-sea sediment containing microorganisms added to sterile sea water)
3. Deep-ocean sediment — Mud 1
4. Deep-ocean sediment — Mud 2 (another set of specimens was exposed in this sediment 5 months later)

The deep-ocean sediment samples were obtained from a depth of 5,300 feet approximately 75 nautical miles southwest of Port Hueneme (33°46'N and 120°37'W). The samples were obtained with a scoop sampler[8] capable of collecting about 225 cubic inches of surface sediment from a fairly soft bottom.

The insulation resistance measurements were performed with a combination voltmeter and ammeter (Figure 3) designed for measurements from 1.0 microvolt to 1,000 volts, and from 0.1 micromicroampere to 1.0 ampere. The ampere data was later converted to resistance by using Ohm's Law.

A 45-volt battery provided the DC current. This battery, in a 5- by 6- by 9-inch aluminum box with a 1/8-inch thick acrylic top, was connected to the input and output terminals and to a two-way switch. The terminals and switch were secured to the acrylic top to maintain maximum electrical resistance between these connections during testing.

The purpose of the two-way switch was to reverse the polarity on each test specimen so that positive and negative readings could be obtained and averaged. Also, any insulation deterioration could be detected by noting the difference in the positive and negative readings produced by the electrolytic action of sea water. Thus any potential produced acts as a battery in series with the measuring circuit and produces the difference in the readings. Figure 4 is a schematic. Resistances were measured in a constant relative humidity (20%) and constant temperature (22.8°C).

Table I. Materials Used in the Formulation of Insulating Materials Tested

Test Specimens	Basic Materials	Plasticizer	Fillers	Antioxidant
Polyethylene	Union Carbide No. 2005 (standard polyethylene insulation)	—	—	—
Polyvinyl Chloride (PVC)	B. F. Goodrich Geon 8801	—	—	—
GR-S (SBR) rubber	Naugapol 1016 and 1018	cumarone-indene resin and micro-crystalline wax	hard clay and water-ground whiting	polymerized trimethyl dihydroquioline
Silicone rubber	Union Carbide No. K1347	—	—	—
Neoprene rubber	Neoprene Type W	light process oil and petroleum	hard clay	4, 4 thiobis (6-tert-butyl m-creosol)

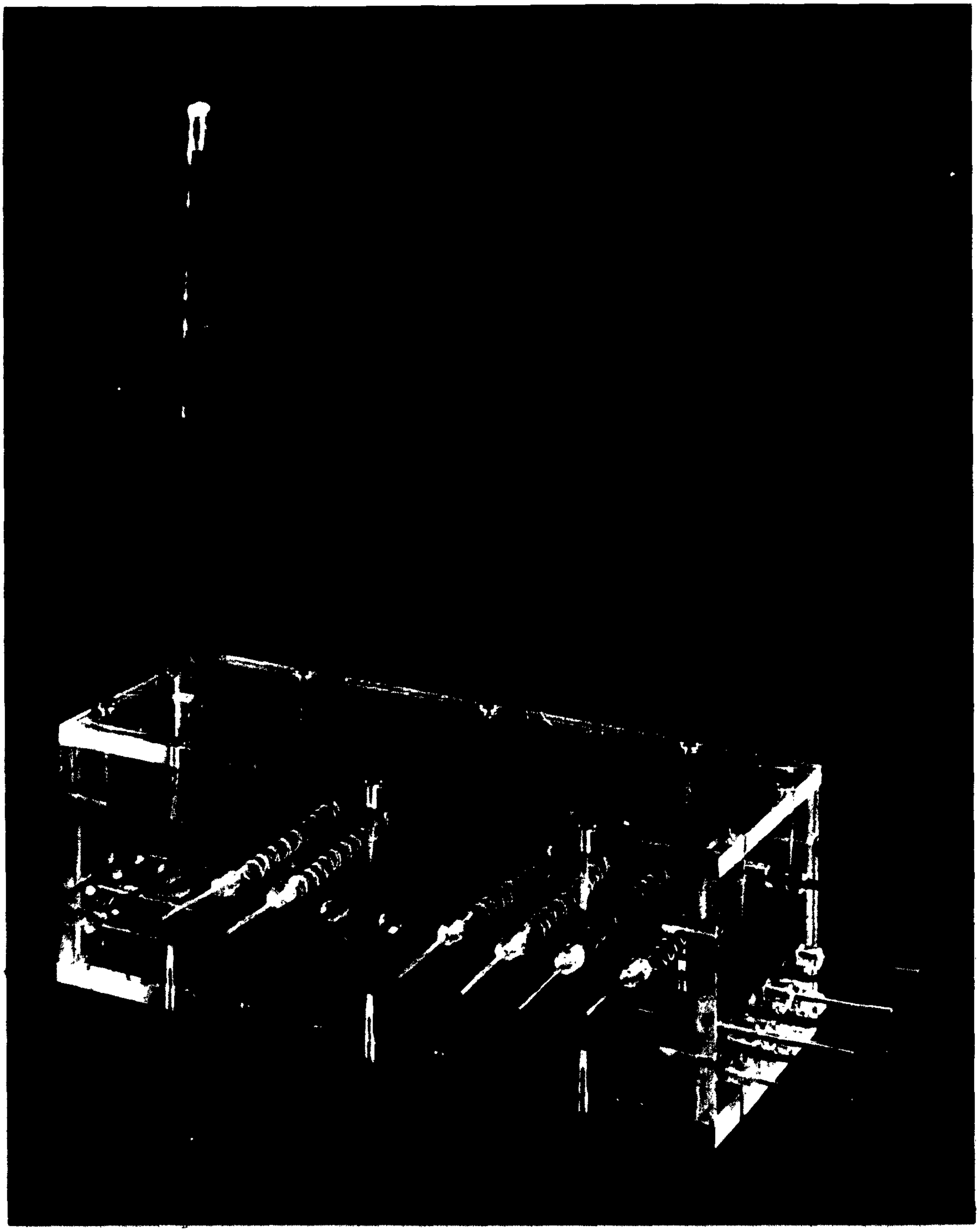

Figure 1. Arrangement of insulating materials in acrylic container.

Figure 2. Specimens in container with sea water and deep-ocean sediment, after 4 months. Note black hydrogen sulfide produced by marine sulfate-reducing bacteria.

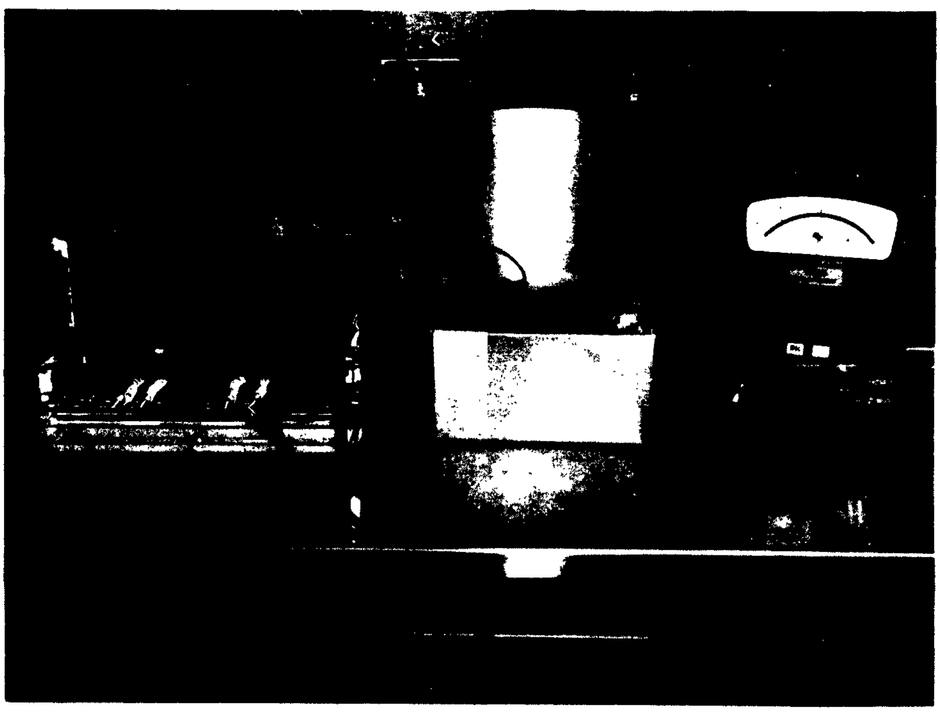

Figure 3. Test setup for measuring insulation resistance of specimens in sterile sea water.

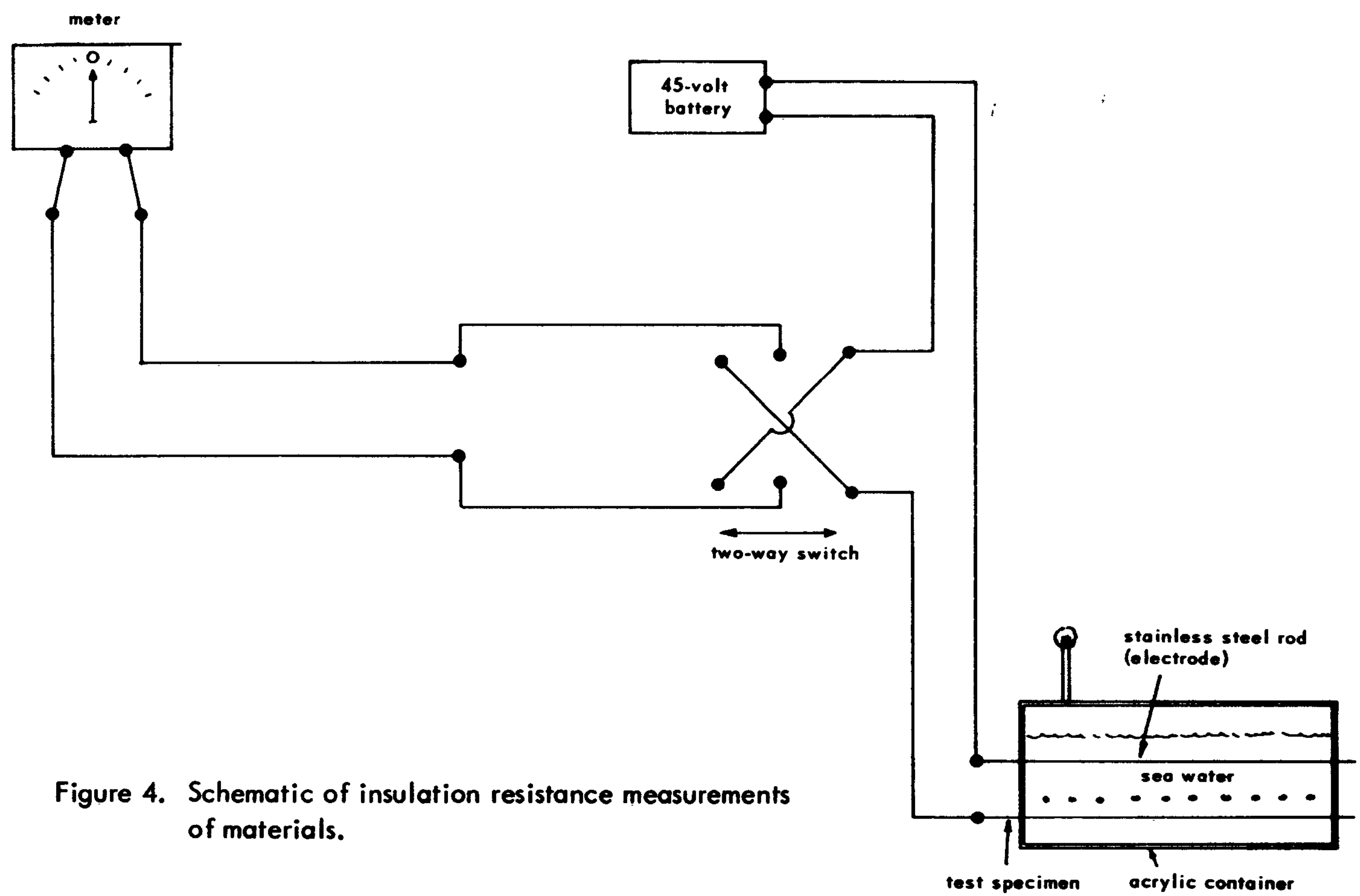

Figure 4. Schematic of insulation resistance measurements of materials.

Concurrently with insulation resistance tests, specimens were subjected to voltage breakdown tests. Duplicates of each material were bent into "U" shape and placed in glass jars; the cut ends protruded through the cover of the jars (Figure 5). The control was filled with sterile sea water; the other was filled with deep-sea sediment containing microorganisms. Periodically, 1,000 volts AC was applied to each specimen for 10 seconds (Figure 6). When not being tested, the specimens in plastic and glass containers were stored in a constant-temperature cabinet at 15°C.

All materials were exposed for 21 months. At the end of this exposure period, a voltage breakdown test was also performed on each of the specimens in the acrylic containers.

Bacteriological analyses of the sediment samples and sterile sea water samples were made before and after the exposure study (Table II). The medium used to determine the aerobic bacterial population consisted of:[9]

Bacto-peptone	5.0 gm
Ferric phosphate	0.1 gm
Yeast extract	1.0 gm
Aged sea water	1,000 ml
Bacto-agar	20 gm
pH	7.5

In addition to the bacteriological analysis, the pH and oxidation-reduction potential of the sea water and sediment samples in the acrylic containers were measured (Table II). Table III summarizes environmental data from the general area where the sediment samples were collected at a depth of 5,300 feet.[10, 11]

RESULTS OF ELECTRICAL MEASUREMENTS

Insulation Resistance

To interpret the relative amount of microbial deterioration of various materials, the rate of decline of insulation resistance values of the test specimens was compared with that of control specimens.

Polyethylene (Figure 7). Polyethylene performed very satisfactorily for about 14 months, after which the insulation resistance values of the specimens declined very sharply at the same rate. The polyethylene was resistant to microbial attack. The decline in insulation resistance values after 14 months was due to water absorption.

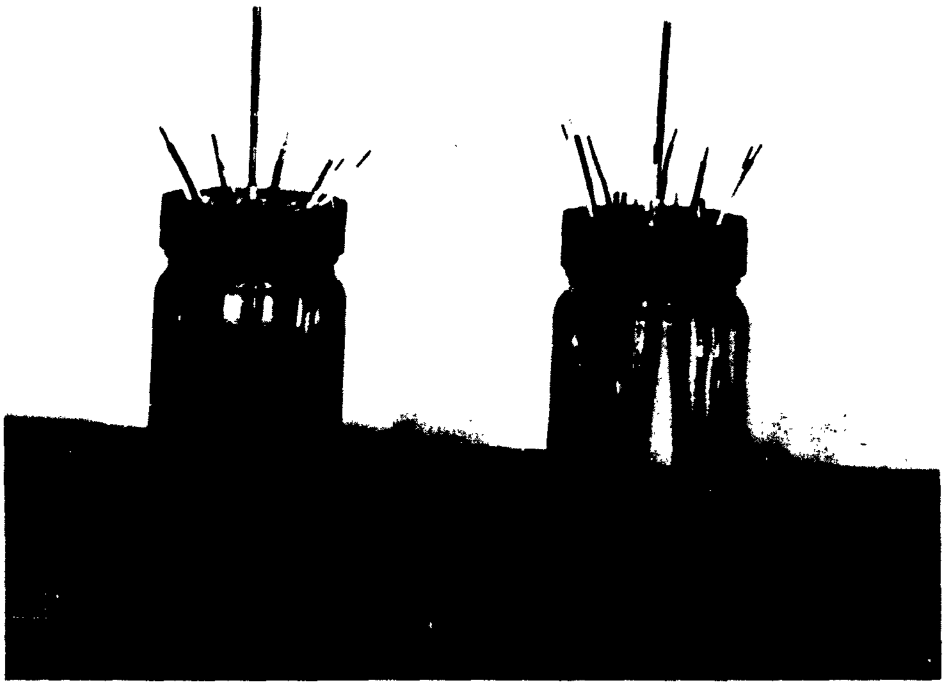

Figure 5. Arrangement of specimens for voltage breakdown tests.

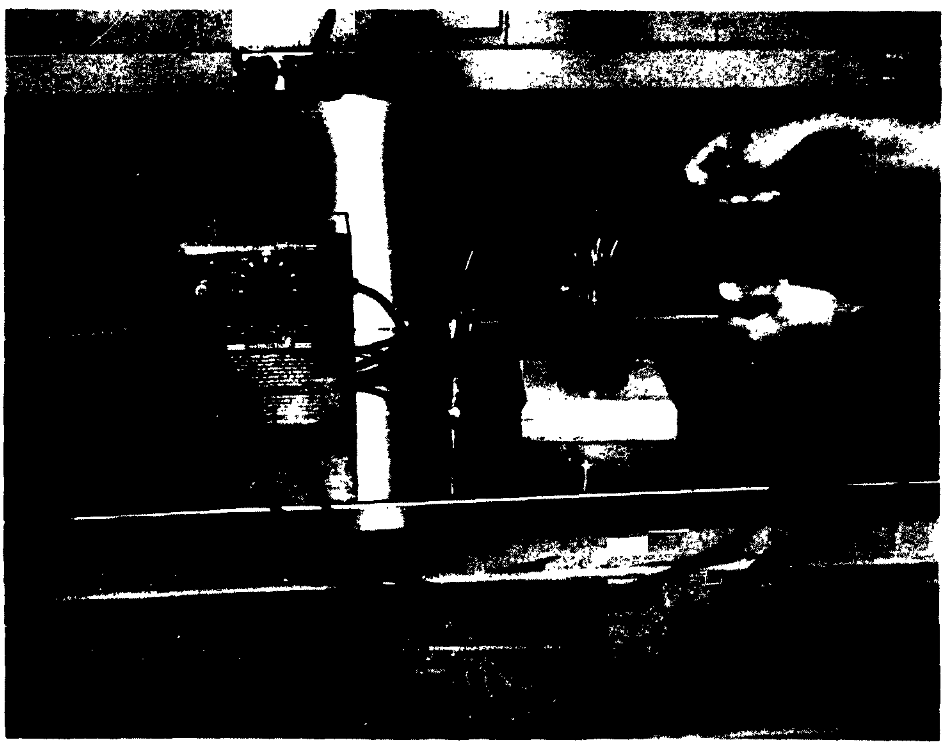

Figure 6. Test setup for voltage breakdown tests of specimens in deep-ocean sediment and sterile sea water.

Table II. Bacteriological Analysis, pH and Oxidation-Reduction Potential Measurements of Sea-Water and Sediment Samples

	At Start of Test				After Termination of Test			
	Sterile Sea Water	Sea Water and Sediment	Sediment		Sterile Sea Water	Sea Water and Sediment	Sediment	
			Mud 1	Mud 2			Mud 1	Mud 2
Number of Aerobic Bacteria	0	6,000 per ml of sea water	25,000 per gm of sediment (wet)		5 per ml of sea water	50,000 per ml of sea water	650,000 per gm of sediment (wet)	
pH Value	8.0	8.0	8.0[1]		7.8	7.7	7.6[1]	7.6[1]
Oxidation-Reduction Potential (mv)[5]	+110	+130	+130[2]	+130[2]	+100	+130	+110[3] +60[2] +20[4]	+130[3] +120[2] +80[4]

Measurements taken:

1/ In sea water above sediment
2/ In center of sediment
3/ At sediment surface
4/ At sediment bottom
5/ Platinum electrode used

Table III. Deep-Sea Environment at a Depth of 5,300 Feet

Location	33°46'N 120°37'W
Water temperature	2.53°C
Hydrostatic pressure	2,335 psi
Dissolved oxygen concentration	1.26 ml/l
Hydrogen-ion concentration (pH)	7.44
Oxidation-reduction potential (E_h)	+215
Salinity	34.56 ppt
Current	less than 0.1 knot
Biological activity in sediment	variety of microorganisms and mud-dwelling animals
Sediment	green mud containing glauconite, Foraminifera tests, quartz and other materials

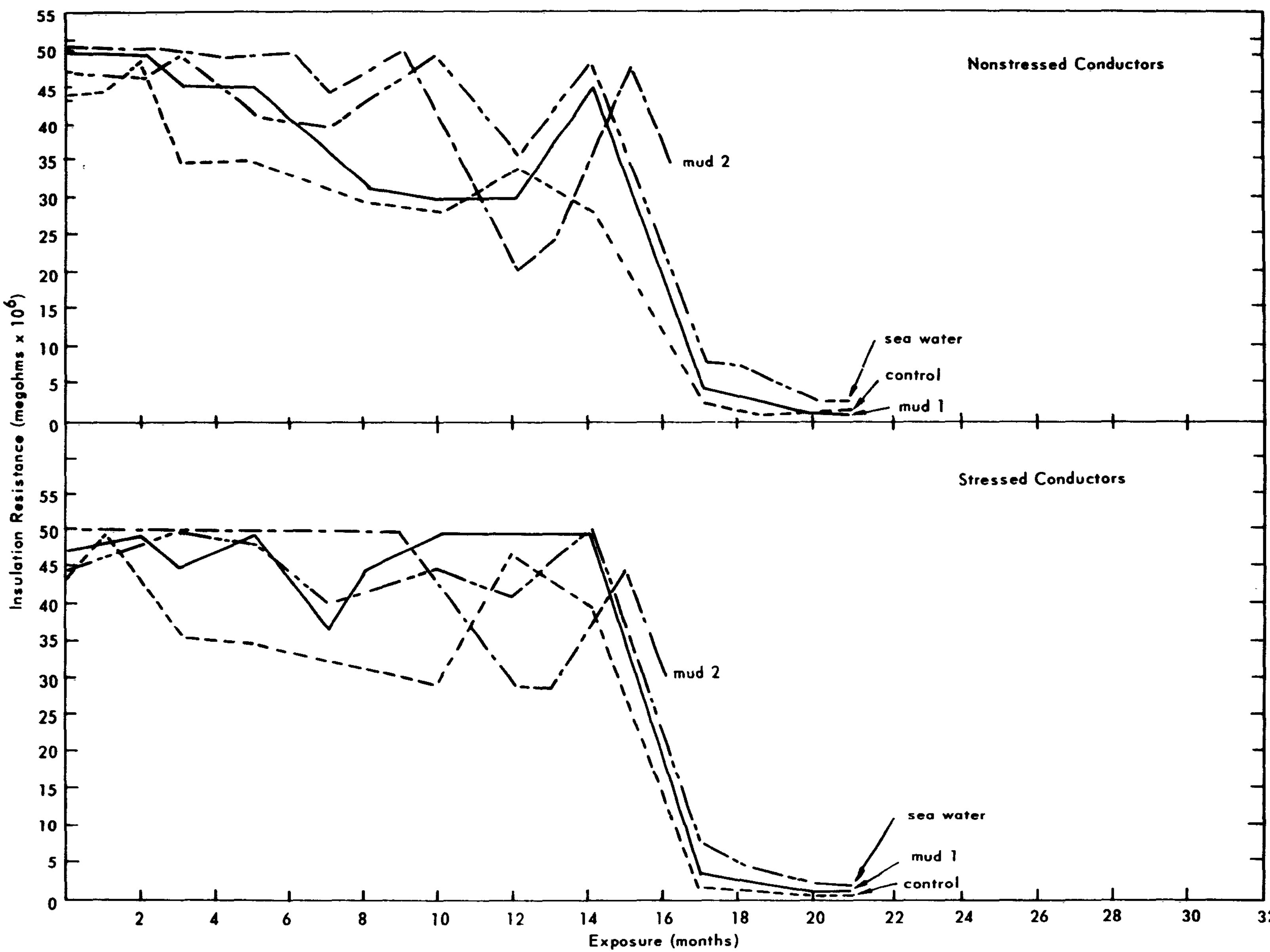

Figure 7. Insulation resistance of polyethylene.

Polyvinyl Chloride (Figure 8). Polyvinyl chloride (PVC) also performed very satisfactorily for 14 months. Then, however, instead of rapidly declining like the polyethylene values, insulation resistance declined gradually, except for one specimen. This was a stressed specimen, exposed in sea water, and its resistance values declined very sharply. In general, the PVC was fairly resistant to microbial attack and to water absorption.

GR-S Rubber (Figure 9). All the stressed specimens of GR-S rubber were fairly resistant to microbial attack and to water absorption. The nonstressed specimens exposed in sea water and in Mud 1 failed after 2 months due to microbial attack. However, one pair of nonstressed specimens exposed in Mud 2 was fairly resistant to microbial attack and to water absorption.

Silicone Rubber (Figure 10). Silicone rubber performed satisfactorily for about 12 months; then the insulation resistance values started to decline fairly rapidly. The decline was caused by slight bacterial attack and by water absorption. In general, silicone rubber insulation was fairly resistant to microbial attack.

Neoprene (Figure 11). The insulation resistance measurements of neoprene specimens clearly differentiated between microbial deterioration and water absorption. The resistance values of control specimens in sterilized water remained at constant level throughout the exposure period, indicating that neoprene is highly resistant to water absorption in the absence of microorganisms. But the resistance values of neoprene specimens in sea water and in sediment dropped quite sharply after about 2 months, indicating failure caused by microbial attack.

Voltage Breakdown

The periodic 10-second application of 1,000 volts AC to each specimen revealed the physical deterioration of the insulating materials. Two neoprene specimens exposed to deep-ocean sediment failed after 14 months. But the neoprene specimens in sterile sea water did not fail in that time, nor did other specimens exposed for the full 21 months in sediment or in sterile sea water.

The insulating materials in the acrylic containers were subjected to a single voltage breakdown test following the insulation resistance measurements after 21 months. There was voltage breakdown in those neoprene test specimens exposed in sea water containing microorganisms and in the two sediments. However, there was no voltage breakdown in neoprene specimens exposed in sterile sea water medium. Nor did voltage breakdown occur in polyethylene, PVC, GR-S rubber, and silicone rubber specimens in mud or sea water.

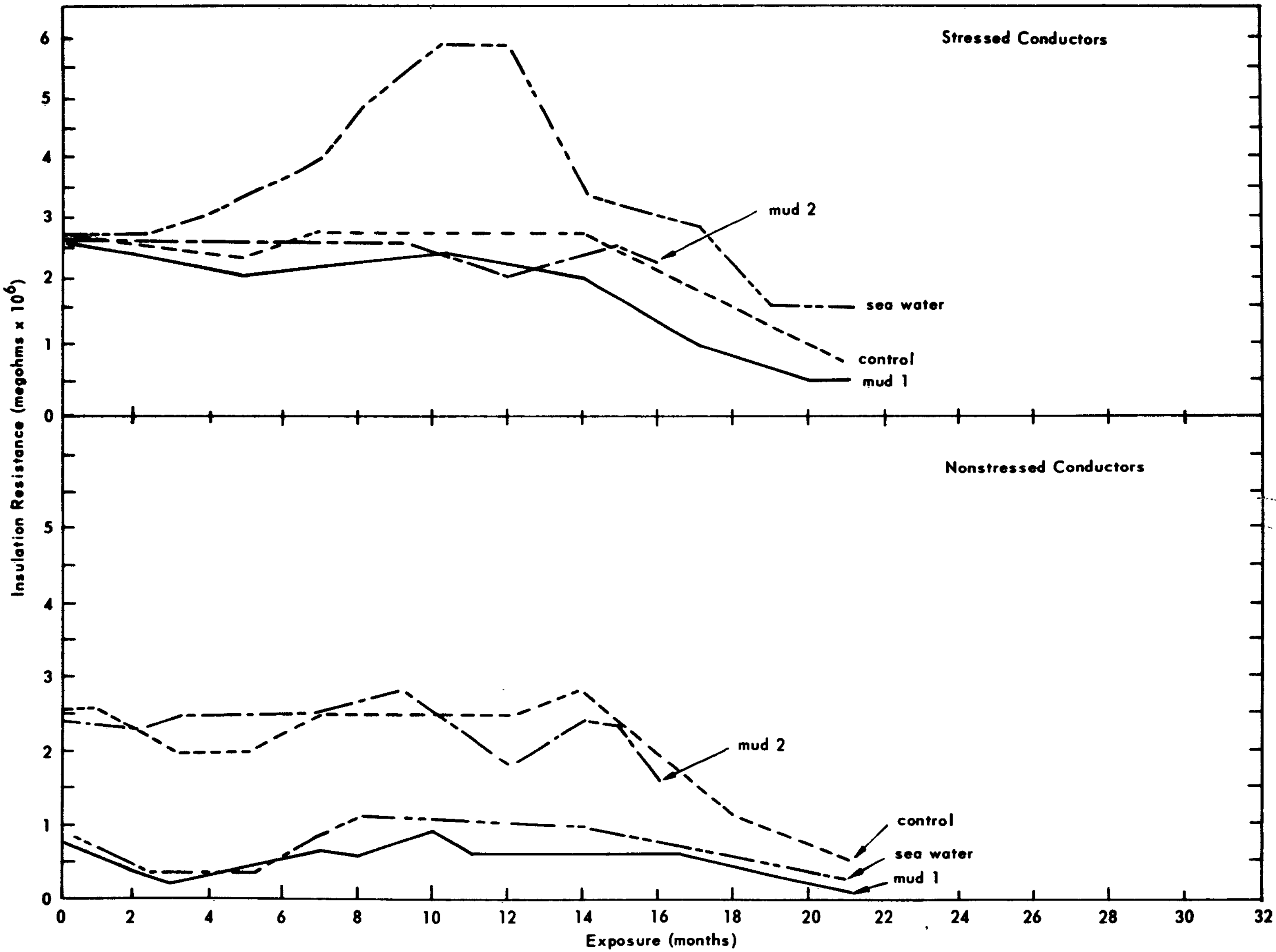

Figure 8. Insulation resistance of polyvinyl chloride (PVC).

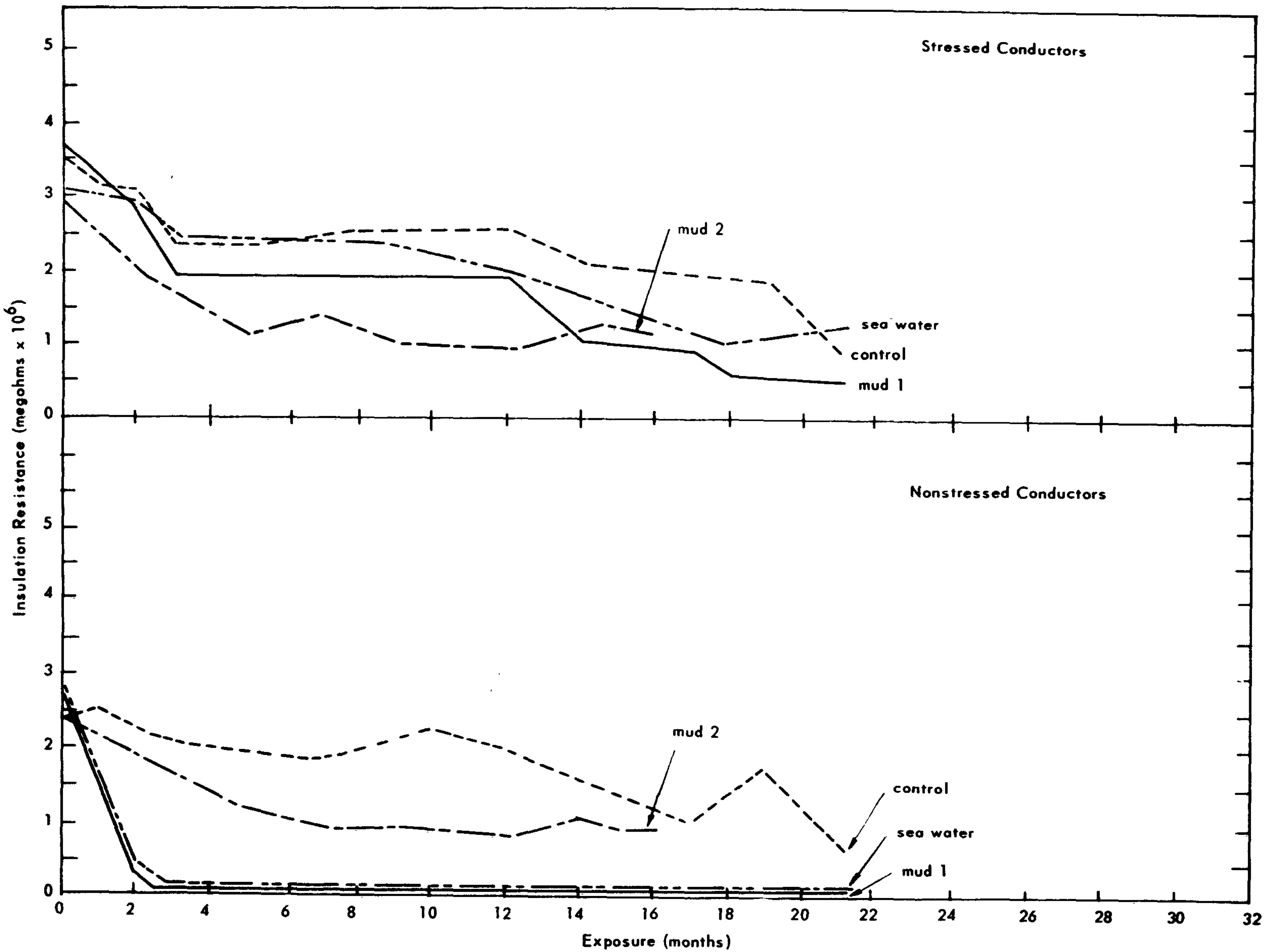

Figure 9. Insulation resistance of GR-S rubber.

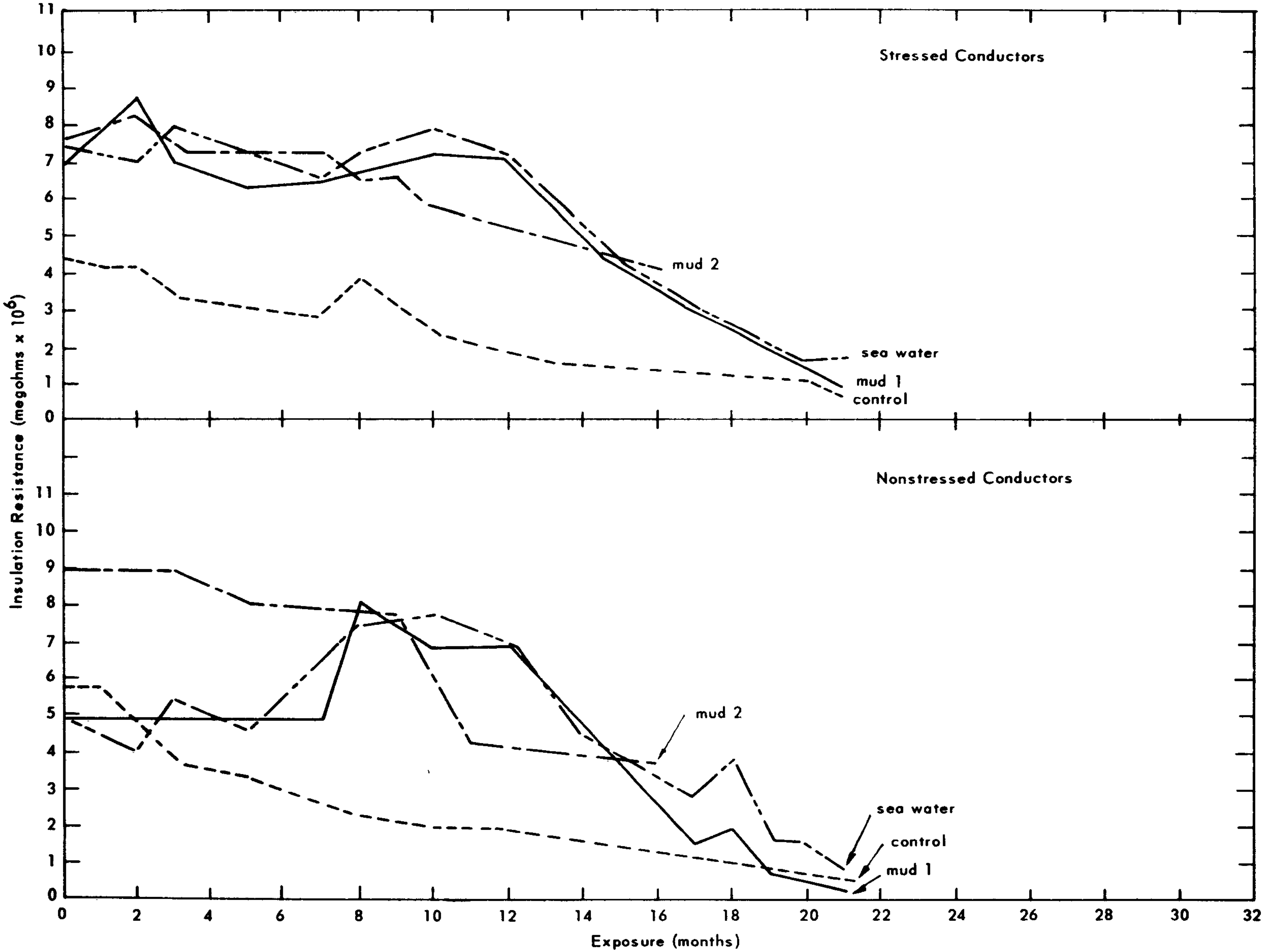

Figure 10. Insulation resistance of silicone rubber.

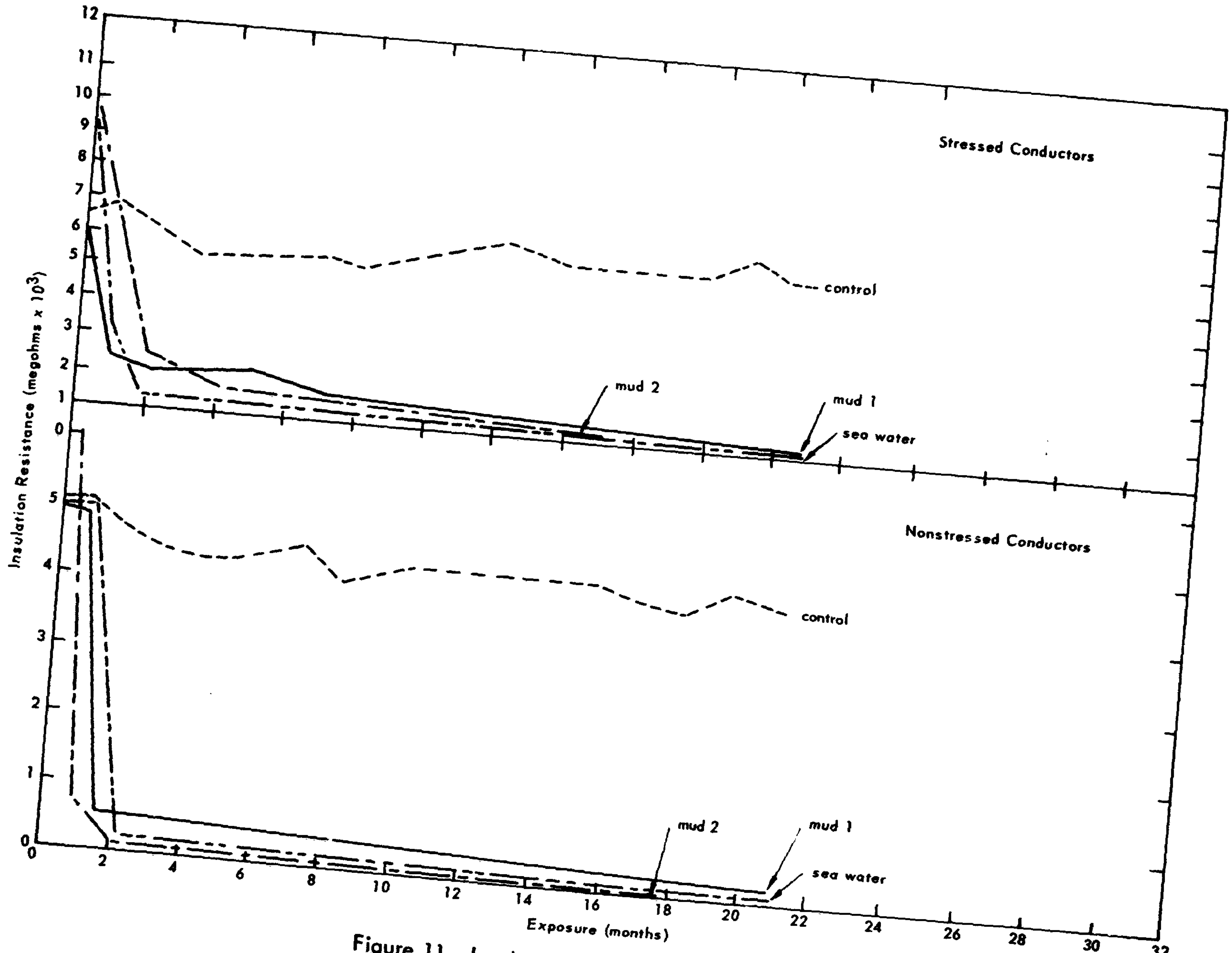

Figure 11. Insulation resistance of neoprene.

MICROSCOPIC EXAMINATION

After termination of the exposure study, the specimens were cut and removed from the acrylic container, washed in tap water, dried, and examined under a stereoscopic microscope. There was no visible surface cracking or other physical damage. However, the sediment-exposed PVC insulating material had changed from white to jet black due to the activity of sulfate-reducing bacteria. The lead stabilizers in PVC had combined with the hydrogen sulfide (H_2S) to form black lead sulfide. In a marine exposure study,[12] it was reported that this discoloration did not change the mechanical properties of the PVC material. The PVC specimens in sterile sea water or in sea water and sediment were not discolored.

The sulfate-reducing bacteria which produced the hydrogen sulfide are anaerobic bacteria. They obtain their energy by the reduction of sulfates and sulfites in water in the absence of free oxygen. The end product of their metabolic process is hydrogen sulfide.

A small amount of hydrogen sulfide was first noted near the bottom of the sediment about 2 months after the exposure test began. Within 6 months, it had covered three-fourths of the sediment in the acrylic container, so that stressed and nonstressed specimens were exposed to it. The top 1-inch layer of the sediment was free of hydrogen sulfide due to the presence of free oxygen.

SUMMARY AND DISCUSSION

Information about the relative resistance of insulating materials to deep-ocean microbial deterioration can be obtained by the insulation resistance (non-destructive) and voltage breakdown (destructive) tests. The average insulation resistance values of the stressed and nonstressed control materials before the test were polyethylene (48 million megohms), silicone rubber (5 million megohms), GR-S rubber (3 million megohms), PVC (2.5 million megohms), and neoprene (5,000 megohms). After 21 months exposure, the average insulation resistance values had dropped to about 1 million megohms for polyethylene, silicone rubber and GR-S rubber. The value for neoprene had remained constant at about 5,000 megohms throughout the exposure period.

Neoprene was highly susceptible to microbial deterioration, but it was highly resistant to water absorption in the absence of microbes. The 15-mil-thick insulated neoprene conductors exposed to microorganisms were not usable after 14 months exposure, but the control materials were usable even after 21 months exposure.

Polyethylene was highly resistant to microbial deterioration, but after 14 months exposure it was highly susceptible to water absorption. Silicone rubber, GR-S rubber, and PVC were all fairly resistant to both microbial deterioration and water absorption, and they were still usable after 21 months. During electrical

measurements on polyethylene, PVC, GR-S rubber, and silicone rubber, it was very difficult to obtain a stable insulation resistance value with the meter because the electrical resistance of these materials was very high (in the micromicroampere range). Thus, the insulation resistance values obtained for these materials are only relative, and no attempt was made to compare the materials with specifications.

When the sterile sea water was analyzed at the end of the exposure period, only one bacterial species, in very low numbers, was found. Shortly before exposure ended, a plastic tube attached to the lid of the acrylic container broke off during handling, and an airborne bacterium could have contaminated the sterile sea water at that time.

The knowledge obtained about the behavior of various insulating materials in this study has practical applications. For example, a superior electrical cable for long-term use in a marine environment could be developed by covering neoprene rubber, which is resistant to water absorption in the absence of microorganisms, with polyethylene, which is resistant to microbial deterioration. For deep-ocean applications, pressure and other factors may change this.

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Of the five materials, neoprene rubber was highly resistant to water absorption in the absence of microbes but was very susceptible to microbial deterioration. Polyethylene was highly resistant to microbes, but after 14 months it was very susceptible to water absorption. Silicone rubber, GR-S rubber, and polyvinyl chloride were fairly resistant to both microbes and water.

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Damage	8					
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Plastics	9		7			
Electrical insulation	9,4		9,4			
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